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PATENT  
Customer No. 22,852  
Attorney Docket No. 05725.0401-01

### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	)	
Roland DE LA METTRIE et al.	)	Group Art Unit: 1751
Application No.: 10/645,881	)	Examiner: Not Yet Assigned
Filed: August 22, 2003	)	
For: COMPOSITION FOR THE	)	
OXIDATION DYEING OF KERATIN	)	
FIBRES AND DYEING PROCESS	)	
USING THIS COMPOSITION	)	

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

#### DECLARATION UNDER 37 C.F.R. 1.132

I, Grégory PLOS, declare and state that:

1. I am a French citizen, residing at Gluck Heim Yoga # 206, 2-12-4 Yoga Setagaya-Ku, 158-0097 TOKYO.
2. I have been awarded a degree in Chemical Engineering from the Institut National Agronomique de Paris-Grignon.
3. I have been employed by L'ORÉAL since 1997 and I am presently a Research Engineer in the Hair Dyeing Applied Research Department at L'ORÉAL. During my employment at L'ORÉAL, I have been engaged in applied research and development regarding hair dyeing and compositions for the treatment of hair.

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4. Given my education and experience, particularly in the area of hair dyeing and compositions for the treatment of hair, I consider myself able to provide the following testimony based on the following additional experiments for U.S. Patent Application No. 10/645,881, conducted by me or under my direct supervision.

## 5. COMPARATIVE TESTING

### **EXAMPLES A AND B: COMPARING INVENTIVE AND NON-INVENTIVE COUPLERS**

Comparative testing was performed with inventive composition B, containing 2-methyl-5-N-( $\beta$ -hydroxyethyl)amino phenol as a coupler, and comparative composition A, which corresponds to a composition according to EP 0 716 846 ("Yamahatsu"), containing m-aminophenol as a coupler. The results of this comparative testing show that inventive composition B is unexpectedly superior to comparative composition A.

#### **I. Compositions**

The following compositions A and B were prepared.

	<b>Composition A (corresponding to EP 0 716 846)</b>	<b>Composition B (invention)</b>
Para-phenylenediamine	0.162 g	0.162 g
Para-aminophenol	0.163 g	0.163 g
Meta-aminophenol	0.327 g	-
2-methyl-5-N-( $\beta$ -hydroxy-ethyl)aminophenol	-	0.501 g
Uric acid	1 g	1 g
Uricase	1 g	1 g
Ethanol	1 g	1 g
2-amino 2-methyl propanol	q.s. pH 9.5	q.s. pH 9.5
Demineralized water	q.s.p. 100 g	q.s.p. 100 g

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## II. Comparative Test Methodology

Each of the resulting mixtures A and B was applied onto locks of natural and permed hair containing 90% white hair. After 30 minutes at room temperature, the hair was rinsed with water, washed with a standard shampoo, rinsed again and dried.

## III. Color Determination

The color of the hair was determined by using the L\*a\*b\* system, with a MINOLTA CM3600d® spectrophotometer (specular components included, light D65, angle 10°). According to this system, L\* indicates the lightness of the color of the hair. The chromaticity coordinates are expressed by the parameters a\* and b\*, a\* indicating the axis of red/green shades and b\* the axis of yellow/blue shades. The chroma value C\* results from the following formula:

$$\sqrt{(a^{*2} + b^{*2})}$$

A higher value of C\* corresponds to more chromatic hair color.

## IV. Results

The results are expressed in the following tables:

Natural hair	a*	b*	C*
Composition A (comparative)	3.2	11.3	11.7
Composition B (inventive)	10.5	11.8	15.8

Permed hair	a*	b*	C*
Composition A (comparative)	3.5	11.2	11.7
Composition B (inventive)	17.2	14.1	22.3

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## V. Analysis and Conclusion

The results tabulated above show that for both natural and permed hair, the composition of the present invention provides a coloration that is more chromatic ( $C^* = 15.8$  for natural hair,  $C^* = 22.3$  for permed hair) than obtained with the composition according to Yamahatsu, EP 0 716 846 ( $C^* = 11.7$  for both natural hair and permed hair).

Based on my education and experience, particularly in the area of hair dyeing and compositions for the treatment of hair, these results would not have been predictable based on the sole difference between the compositions being the use of m-aminophenol as a coupler in composition A compared with the use of 2-methyl 5-N-( $\beta$ -hydroxyethyl)amino phenol as a coupler according to composition B. The results have statistical significance, and the differences between the  $a^*$ ,  $b^*$ , and  $c^*$  values are greater than the known error according to the above described color determination method. The results are also of practical significance, as greater chromaticity in both natural and permed hair is a desirable and useful characteristic for a hair dyeing composition or method.

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### EXAMPLES C AND D: COMPARING OXIDIZING SYSTEMS

Comparative testing was performed with inventive composition D, comprising the 2-electron oxidoreductase enzyme uricase/uric acid system as an oxidizing system, and comparative composition C, which corresponds to a composition according to U.S. Patent No. 4,065,255 ("Andrillon") and contains hydrogen peroxide as an oxidant. The results of this comparative testing show that inventive composition D is unexpectedly superior to comparative composition C.

#### I. Compositions

The following compositions C and D were prepared.

	Composition C (corresponding to US 4,065,255)	Composition D (invention)
Para-phenylenediamine	0.162 g	0.162 g
Para-aminophenol	0.163 g	0.163 g
2-methyl-5-N-( $\beta$ -hydroxy-ethyl)-aminophenol	0.250 g	0.250 g
Hydrogen peroxide	1 volume	-
Uric acid	-	1 g
Uricase	-	1 g
2-amino 2-methyl propanol	q.s. pH 8.5	q.s. pH 8.5
Demineralized water	q.s.p. 100 g	q.s.p. 100 g

#### II. Comparative Test Methodology

Each of the resulting mixtures C and D was applied onto locks of natural and moderately bleached hair containing 90% white hair. After 30 minutes at room temperature, the hair was rinsed with water, washed with a standard shampoo, rinsed again and dried.

### III. Color and Selectivity Determination

The color of the hair was determined using the L\*a\*b\* system as described above. Also determined was the parameter  $\Delta E$ , which is the color variation between a colored lock of natural hair and a colored lock of moderately bleached hair.  $\Delta E$  is obtained from the following formula:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2}$$

wherein L\* indicates the lightness and a\* and b\* are the chromaticity coordinates of the colored lock of natural hair whereas L<sub>0</sub>\* indicates the lightness and a<sub>0</sub>\* and b<sub>0</sub>\* are the chromaticity coordinates of the colored lock of moderately bleached hair, as obtained from the above color determination method. A lower value of  $\Delta E$  corresponds to less selective hair coloration.

### IV. Results

The of the color determination results are expressed in the following table:

Natural hair	a*	b*	C*
Composition C	9.7	4.1	10.5
Composition D	11.8	11.4	16.4

The results of the color variation determination are expressed in the following table:

	Natural hair			Weakly bleached hair			
	L*	a*	b*	L*	a*	b*	$\Delta E$
Comp. C	28.0	9.7	4.1	19.0	4.4	1.2	10.8
Comp. D	37.3	11.8	11.4	30.3	14.5	11.5	7.5

#### V. Analysis and Conclusion

The results of the color determination show that a composition according to the present invention (composition D) provides a coloration that is more chromatic ( $C^* = 16.4$ ) than obtained with the composition according to Andrillon, US 4,065,255 (composition C) ( $C^* = 10.5$ ).

The results of the color variation determination show that a composition according to the present invention (composition D) provides a coloration that is less selective than obtained with the composition according to Andrillon, US 4,065,255 (composition C), i.e., inventive  $\Delta E$  of 7.5 versus comparative  $\Delta E$  of 10.8.

Based on my education and experience, particularly in the area of hair dyeing and compositions for the treatment of hair, these results would not have been predictable based on the sole difference between the compositions being the use of hydrogen peroxide as an oxidant in composition C compared with the use of the 2-electron oxidoreductase enzyme uricase as an oxidant according to composition D. The results have statistical significance, and the differences between the  $a^*$ ,  $b^*$ , and  $c^*$  values and  $\Delta E$  are greater than the known error according to the above described color and selectivity determination methods. The results are also of practical significance, as

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greater chromaticity and reduced selectivity are desirable and useful characteristics for a hair dyeing composition or method.

6. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 2004/05/11

By: Gregory PLOS.  
Grégory PLOS